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L5: Entry 1 of 30 File: USPT Nov 5, 2002

DOCUMENT-IDENTIFIER: US 6476215 B1
TITLE: Ink jet method of spotting a probe and manufacturing a probe array

Detailed Description Text (51):
The amino or epoxy group can be introduced to a glass plate as the solid support by, first treating the surface of the glass plate with an alkali solution such as potassium hydroxide and sodium hydroxide to expose hydroxyl groups (silanol groups) to the surface, and then reacting a silane coupling agent containing a silane compound to which an amino group has been introduced (for example, N-.beta.-(aminoethyl)-.gamma.-aminopropyltrimethoxysilane, etc.) or a silane compound to which an epoxy group has been introduced (for example, .gamma.-glycidoxypropyltrimethoxysilane, etc.) with a hydroxyl group of the surface of the glass plate. To introduce maleimido groups to the surface of the glass plate, the amino groups introduced by the above method are reacted with N-maleimidocaproyloxy succinimide or succinimidyl-4-(maleimido phenyl)butyrate, etc.

Detailed Description Text (54):
When a resin plate is used as a solid support, hydroxyl groups, carboxyl groups, or amino groups can be introduced to the surface of resin substrate according to the method described in Chapter 5 of "Organic Thin Films and Surface", Vol. 20, Academic Press. Alternatively, after introducing hydroxyl groups by this method, as is shown for the glass plate mentioned above, amino groups or epoxy groups can be introduced by using a silane compound having amino group or epoxy group. Further a maleimido group can be introduced. Functional groups can be introduced either before or after the matrix pattern is formed on a solid support. Before matrix pattern formation, a reaction solution required for introduction of a functional group can be supplied to a solid support surface by spin coating or dip coating, etc. After matrix formation, a reaction solution can be supplied to each well by the ink jet method, etc.

Detailed Description Text (76):
A glass plate of 1 inch.times.1 inch was placed in a rack and immersed in an ultrasonic washing detergent overnight. After ultrasonic washing in the detergent for 20 minutes, the detergent was removed by rinsing with water. After rinsing with distilled water, ultrasonic treatment was performed in a container containing distilled water for 20 minutes. The glass plate was immersed for 10 minutes in a 1 N sodium hydroxide solution preheated to 80.degree. C. Then, the plate was washed with water and distilled water to prepare a glass plate for a probe array.

Detailed Description Text (194):
(2) In the same manner as in (1) of Example 7, an 1 wt % aqueous solution of a silane coupling agent (Product name: KBM403, from Shin-Etsu Chemical Co., Ltd.), which contains a silane compound having an epoxy group (.gamma.-glycidoxy propyl trimethoxysilane), was stirred at room temperature for 1 hour to hydrolyze methoxy groups of the silane compound molecule. Then the solid support prepared in the above description (1) was immersed in this solution at room temperature for 30 minutes and washed with distilled water, and after the remaining water was removed by nitrogen gas flow, it was baked at 120.degree. C. for 5 minutes to introduce epoxy groups to the bottom surface of the wells. At this point, the black matrix surface was water-repellant since its contact angle to water was 95.degree., and the bottom of the wells was hydrophilic since its contact angle to water was 33.degree.. Thus, introduction of epoxy groups to the bottom surface of the wells is also made possible by treating the BM-formed solid support with a silane coupling agent.

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L5: Entry 9 of 30 File: USPT Oct 24, 2000

DOCUMENT-IDENTIFIER: US 6136962 A
TITLE: Covalent attachment of unmodified nucleic acids to silanized solid phase surfaces

Brief Summary Text (8):
Many hybridization assays require the immobilization of one component. Nagata et al. described a method for quantifying DNA which involved binding unknown amounts of cloned DNA to microtiter wells in the presence of 0.1 M MgCl.sub.2 (Nagata et al., FEBS Letters 183: 379-382, 1985). A complementary biotinylated probe was then hybridized to the DNA in each well and the bound probe measured colorimetrically. Dahlen, P. et al. have discussed sandwich hybridization in microtiter wells using cloned capture DNA adsorbed to the wells (Dahlen, P. et al., Mol. Cell. Probes 1: 159-168, 1987). An assay for the detection of HIV-1 DNA using PCR amplification and capture hybridization in microtiter wells has also been discussed (Keller, G. H. et al., J. Clin. Microbiol. 29: 638-641, 1991). The NaCl-mediated binding of oligomers to polystyrene wells has been discussed by Cros et al. (French patent no. 2,663,040) and very recently by Nikiforov et al. (PCR Methods Applic. 3: 285-291, 1994). The cationic detergent-mediated binding of oligomers to polystyrene wells has very recently been described by Nikiforov et al., Nucleic Acids Res. 22: 4167-4175.

Detailed Description Text (27):
An important feature of the present invention is the hydrophobic nature of silanes. Because of this feature, it is possible for an aqueous solution to form extremely well defined beads on the surface of any solid support coated with mercaptosilane. With an automated delivery system, such as a Hamilton robot or ink-jet printing method, it is possible to form a very complex array of oligonucleotide probes on a mercaptosilane coated glass slide. Such methods can deliver nano to pico-liter size droplets with sub-millimeter spacing. Because the aqueous beads are extremely well defined, it is possible to create an array with an extremely high density of oligonucleotide probes. Thus, it is possible to create arrays having greater than about 10,000 probe droplets/cm.sup.2.

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L5: Entry 11 of 30 File: USPT May 9, 2000

DOCUMENT-IDENTIFIER: US 6060237 A
TITLE: Devices and methods for optical detection of nucleic acid hybridization

Brief Summary Text (14):
system allows for the chemical synthesis of a strand of nucleic acids on the surface. The primary advantage of the linker is that it is stable to an ammonia treatment which is required in the synthesis of the polynucleotide. A hexaethylene glycol spacer is incorporated into the linker which attaches to the glass through a glycidoxypropyl silane which terminates in a primary hydroxy group. The silane is condensed onto silane groups on a solid support. Additional cross-linking may be obtained by introducing water so that the epoxide group is cleaved to a diol. An acidic solution facilitates this process. The length of the linker may be varied by changing the spacer to ethylene glycol, pentaethylene glycol, etc.

Detailed Description Text (11):
In one example, the optical substrate is formed from a silicon crystal which is grown and extruded to 4 inches in diameter and then diamond sawed to form a wafer. The wafers are treated with chemical etchants to smooth the surface and reduce flaws. The wafers are lapped or ground with aluminum oxide, titanium oxide, or silicon carbide particles in a talc slurry. The initial grain size is large and successively smaller particle sizes are used to produce an increasingly smoother surface. Both sides of the wafer are subjected to this process. The final lapping process leaves a very diffusely reflective surface. Wafers may be further processed with chemical or plasma etching to modify the diffuse reflecting characteristic of the substrate. Once the wafers are lapped, they are cleaned using the following process or a known modification thereof: the wafers are sonically cleaned with a cationic detergent, followed by a rinse with 18 megaohm water. Then they are cleaned with an anionic detergent, followed by a rinse in 18 megaohm water. They are ultrasonically cleaned with an aqueous ammonia solution made of 370 ml of 30% H.sub.2 O.sub.2, 250 ml of aqueous ammonia and 9 gallons of water, and are rinsed in a cascade of water with the final rinse being with 0.1 micron filtered water. They are then spin-dried and are ready for optical coating. An alternative to this procedure is the "RCA Clean" described in Polymer Surfaces and Interfaces, edited by W. J. Feast and H. S. Munro, John Wiley and Sons, New York, N.Y., page 212, 1987.

Detailed Description Text (67):
DNA capture probe was coated onto these wafer surfaces from a solution containing 50 mM sodium citrate, pH 6.0, 0.1 mg/ml carrier DNA, sheared herring sperm DNA, and 600 .mu.M biotinylated DNA, 26-mer. The probe sequence was 5'-CGCTAATATCAGAGAGATAACCCAC-3'. Wafers were incubated in this solution overnight at 4.degree. C. Wafers were removed from the solution and washed with 1.times. phosphate buffered saline containing 0.2% Tween 20.TM. detergent (PBS/Tween). The wafers were then coated in a BSA (bovine serum albumin) solution for 3 hours at 65.degree. C. The wafers were then rinsed with PBS/Tween detergent.

Detailed Description Text (68):
To measure the amount of biotinylated DNA adsorbed to the surface, the wafers were incubated for 30 minutes with a solution containing streptavidin conjugated to horseradish peroxidase (Immunology Products) was diluted 1:250 in 50 mM MOPS, pH 7.0, containing 3% alkaline treated casein, 0.2% TWEEN20 detergent, and 0.5% Proclin 300 (an anti-bacterial agent). Wafers were then washed with deionized water and dried under a stream of nitrogen. A drop of TMB precipitating substrate was applied to the

surface and the wafers incubated for 30 minutes at room temperature. Thickness increases (in Angstroms) due to substrate deposition were measured using an absolute ellipsometer (Gaertner) which was normalized to the initial substrate thickness. See FIG. 1. These experiments were repeated using alkaline phosphatase conjugated to streptavidin and BCIP/nitroblue tetrazolium substrate pair. The R-polymer surface (most polar surface used) and the T-polymer surface (least polar surface used) all performed well. These experiments demonstrate that DNA can be successfully immobilized to the surface of the silicon wafer.

Detailed Description Text (71):
Wafers coated with T-polymer (see Example 1) were coated for 56 hours at 4.degree. C. in a solution containing 50 mM sodium citrate buffer, pH 6.0, 5.times. SSC, and 20 .mu.g/ml of the ssDNA capture probe complimentary to M13mp18. The probe sequence was CGCTAATATCAGAGAGATAACCCAC. Probe coated wafers were removed from coating solution and placed into a blocking solution containing 5.times. Denhardt's solution, 0.5% SDS, 1 mg/ml carrier DNA, and 25 mM buffer at pH 6.5. They were incubated 16-18 hours at 4.degree. C. and then rinsed with phosphate buffered saline containing 0.0005% TWEEN20 detergent at pH 7.4. Capture probe coated wafers were hybridized with M13mp18 plasmid overnight at 60.degree. C. in a solution containing 1.times. Denhardt's solution, 0.5% SDS, 25 mM MES, pH 6.5, 0.2 mg/ml carrier DNA, 5.times. SSC, a final concentration of M13mp18 was 500 ng/ml, 1 ng/ml or 100 pg/ml. The final hybridization step occurred under the same solution and incubation conditions as the previous step with a final biotinylated amplifying probe concentration of 92 .mu.M. The amplifying probe contains strand sequence from 6249 to 6273 and was biotinylated at residue 6261. The sequence is GCAGGTCGACTGTAGCAGGATGCCGG. All appropriate controls were performed. Wafers were incubated with a streptavidin alkaline phosphatase conjugate. Precipitating substrate, BCIP/nitroblue tetrazolium, was used to generate an increase in thickness at the surface of the wafer. Thickness increases were measured using an absolute ellipsometer (Gaertner). Results for the experiment are shown in FIG. 2. From this experiment, it was concluded that a sensitivity of 1 ng/ml and potentially as low as 100 pg/ml was achieved. This translates to a copy number of roughly 10.sup.10 for a very un-optimized assay.

L5: Entry 16 of 30 File: USPT Jan 12, 1999

DOCUMENT-IDENTIFIER: US 5858630 A
TITLE: Process for treating a photographic bath containing organic contaminants

Detailed Description Text (17):
When silane film-forming compounds as described previously are used, the lipophilic layer is obtained from a solution of silane in an aprotic anhydrous solvent. This solution is put in contact with the "active" support in the presence of water. By hydrolysis, an Si-O bond is formed between the support and the silane, which immediately grafts the silane onto the support. The homogeneity of the layer is obtained by means of -Si-O-Si- bonds between the silane molecules, as shown in FIG. 1.

Detailed Description Text (18):
In this way a material is obtained with a lipophilic surface, which is mechanically and chemically very strong, and which will trap the tars formed by the organic contaminants originating from the photographic product being processed, such as residues of plasticizers, latex, surfactants, lubricants, organic contaminants of the treatment bath, for example the bath oxidation products, bactericides, detergents and any other compound insoluble in the water present in the baths.

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L5: Entry 26 of 30 File: USPT Jun 2, 1992

DOCUMENT-IDENTIFIER: US 5118607 A
TITLE: Non-aqueous solvent specific binding protein assays

Brief Summary Text (16):
With many of these samples, it may be necessary or desirable to extract lipid soluble material into an organic solvent. Thus, the extract will provide the sample to be used. For extraction, the same solvent as used as the assay medium is not required, so long as the extracting solvent is substantially miscible with the assay medium in the amounts used. Thus, various solvents may be used, which have higher hydrophilicity than the solvents used in the subject assays. In some instances, the compound of interest may be dispersed in an aqueous medium due to the presence of solubilizing compounds such as proteins or detergents. In this instance, so long as the sample is small and can be dispersed in the hydrophobic assay medium without separation, the sample may be used directly.

Brief Summary Text (25):
For heterogeneous assays, the receptor will normally be bound to a solid support. Solid supports may include rods, beads, membranes, vessels, for example, microtiter wells, or the like. Various materials may be involved, such as glass, nylon, Mylar, controlled pore glass, or other support which will not be adversely affected by the assay medium. Immobilization of the receptor may be achieved in a variety of conventional ways, usually employing covalent binding in accordance with conventional techniques. The glass products can be readily activated by heating the glass at elevated temperature, generally in the range of about 400 to 600.degree. C. After cooling the glass is functionalized by reaction with an appropriately functionalized silane. Controlled pore glass needs no activation prior to silane functionalization. Thus, halodialkoxy or trialkoxycarboxyalkyl or -aminoalkyl silanes may be employed. Functionalized supports may be readily conjugated to the receptor using various reagents, such as glutaraldehyde, maleimidobenzoic acid, where the protein has a mercapto group, or where sugars are present on the protein, these may be oxidized to dialdehydes for linkage under reducing conditions to an amine, to form a methyleneamine or imine. After reaction of the specific binding protein to the surface, the surface may then be washed with an aqueous medium, particularly one containing inert .gamma.-globulin. After incubation with .gamma.-globulin containing solution, the surface may then be exhaustively washed with an aqueous medium, particularly phosphate buffered saline.

CLAIMS:

6. The method according to claim 5, wherein said aliphatic hydrocarbon is a straight chain hydrocarbon of from 6 to 16 carbon atoms and said detergent is a sulfo salt.

L5: Entry 27 of 30 File: USPT Feb 18, 1992

DOCUMENT-IDENTIFIER: US 5089299 A
TITLE: Composite ceramic micropermeable membrane, process and apparatus for producing such membrane

Brief Summary Text (42):
Preferably, the treatment with the silane composition results in a monolayer of silicon containing groups. For example, infrared analysis shows that, after treatment with trimethoxy-methylsilane, hydroxy-methoxy-methylsilyloxy groups are present on the support. Assuming a mean surface area per silyl group of 1 nm.sup.2, 10 mg of trimethoxymethylsilane produces a monolayer of about 40 m.sup.2. This means that 1 liter of a 1% silane solution is sufficient for pretreating a support having an effective surface area of 40 m.sup.2.

Brief Summary Text (49):
If the affinity has been adjusted by silane treatment, as described above, a further adjustment in affinity can be made by adding a detergent to the aqueous suspension, making the affinity between the support and the suspension higher again, inducing an attractive interaction between the porous support and the microporous layer, while at the same time the pores of the support are not penetrated excessively with suspended particles.

Detailed Description Text (2):
A tube having an external diameter of 20 mm and a wall thickness of 3 mm, consisting of a mixture of sintered alpha alumina and mullite having a pore size range between 0.1 .mu.m and 5 .mu.m and an average pore radius of 1 .mu.m (determined by mercury porosimetry) with large surface pores of up to 50 .mu.m (observed by electron microscopy), was slowly immersed into a 1% silane solution (Silane PC.sub.12 from Permacol B.V., Holland) in acetone, for 10 minutes, allowing all pores of the tubular support to be filled with the solution. Then, the tube was taken from the solution and dried in the air at 50.degree. C., until constant weight.

Detailed Description Text (5):
A tube having an external diameter of 20 mm and a wall thickness of 2.5 mm, consisting of sintered alpha alumina having an average pore radius of 1.5 .mu.m (determined by mercury porosimetry) with large surface pores of up to 20 .mu.m (observed by electron microscopy), was slowly immersed into a 1% silane solution (Silane PC.sub.12 from Permacol B.V., Holland) in acetone, for 10 minutes allowing all pores of the tubular support to be filled with the solution. Then the tube was taken from the solution and dried in the air at 50.degree. C., until constant weight.

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L5: Entry 30 of 30 File: USPT Oct 18, 1988

DOCUMENT-IDENTIFIER: US 4778461 A
TITLE: Heart valve prosthesis and process for its production

Detailed Description Text (35):
Attrition particles, traces of lubricants etc. may be present on the support ring from the production process. These are removed preferably by a two-stage washing process. The first washing process is carried out with water and detergent, and the second is carried out with an organic solvent or solvent mixture. For this purpose, the known organic solvents are suitable, inter alia, such as acetone, ethyl acetate, ethanol, isopropanol, trichlorethylene or gasoline. The preferred surface-active substances are anionic compounds such as sodium laurylsulfonate.

Detailed Description Text (37):
According to the invention, the process for the production of the novel heart valve prosthesis by the dip-coating process with a cusp material of polyurethane, that is to say above all from the polyether-urethanes described, is carried out in such a way that the washed, etched and rinsed support ring is dipped several times into a polyurethane solution (solvent: dimethylacetamide) containing a crosslinking agent and, in between, is dried each time to such an extent that the film no longer flows. The .gamma.-aminopropyltrisethoxy-(or trimethoxy)-silane already mentioned is the preferred suitable crosslinking agent. When a coating of 30-50 micrometers thickness has been reached, which as a rule requires 3-5 dipping steps (depending on the viscosity and concentration of the solution), the ring is pushed over a dip mould which precisely fits the cusps and which is machined such that the three cusps with commissures are formed integrally, and the entire workpiece is then subjected to further dipping steps until the desired film thickness of the cusps of 80-200 micrometers has been reached. This requires another 8-20 dipping steps, depending on the concentration and viscosity of the solution.